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IS 3025 (Part 01) (1987, Reaffirmed 2003): Method of Sampling and Test (Physical and Chemical) for Water and Wastewater, Part 01: Sampling (First Revision). ICS 13.060.50

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“Knowledge is such a treasure which cannot be stolen”

UDC 628·1/·3 : 543·05

(Fourth Reprint MAY 2008)

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(Reaffirmed 2003)

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 1 SAMPLING

(First Revision)

1. Scope — Prescribes the methods of sampling of water and wastewater for physical and chemical examinations.

2. Terminology — For the purpose of this standard, the definitions given in IS : 7022 (Part 1)-1973 'Glossary of terms relating to water, sewage and industrial effluents: Part 1, and IS : 7022 (Part 2)-1979 'Glossary of terms relating to water, sewage and industrial effluents: Part 2', shall apply.

3. Sampling

3.1 *Filling the Containers* — In the case of samples for the determination of physico-chemical parameters one simple precaution, which is not, however, adequate in all cases, is to fill the flasks completely and stopper them in such a way that there is no air above the sample. This limits interaction with the gas phase and agitation during transport (thus avoiding modifications in carbon dioxide content, and hence variations in pH; hydrogencarbonates are not converted into precipitable carbonates; iron has less tendency to be oxidized, thus limiting colour variations; etc).

Sample containers, whose contents are frozen as part of their preservation, should not be completely filled.

3.2 *Use of Appropriate Containers* — The choice and the preparation of a container can be of major importance. However, it should be remembered that the container in which the sample is stored and the stopper should not:

a) be a cause of contamination (for example, borosilicate or soda-lime glass containers may increase the content of silica or sodium);

b) absorb or adsorb the constituents to be determined (for example, hydrocarbons may be absorbed in a polyethylene container, traces of metals may be adsorbed on the surface of a glass container); and

c) react with certain constituents in the sample (for example fluorides reacting with glass).

It should be remembered that the use of opaque containers or brown (non-actinic) glass containers can reduce the photosensitive activities to a considerable extent.

Blank samples should be taken, preserved and analyzed as a check on the suitability of the choice of container and cleaning procedure.

3.3 *Cleaning of Containers*

3.3.1 *For samples for general chemical analysis*

3.3.1.1 For analysis of trace quantities of chemical constituents of surface or wastewater, it is usual to clean new containers thoroughly in order to minimize possible contamination of the sample; the type of cleaners used and the container material vary according to the constituents to be analyzed.

For general purposes, new glass containers should be cleaned with water and detergents to remove dust and packing material. They should then be cleaned with chromic acid-sulphuric acid mixture before being thoroughly rinsed with distilled water.

Water Sectional Committee, CDC 26; Panel for Methods of Test for Water and Effluents CDC 26 : P1 [Ref : Doc : CDC 26 (8974)]

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It may be desired, for environmental or health reasons, to avoid the use of chromic acid. Alternatively, proprietary cleaning agents may be used, provided it has been established that they do not cause sample contamination.

It should be noted that detergents, possibly containing phosphates, cannot be used if phosphates or surface-active agents are to be determined, nor can chromic acid-sulphuric acid mixture be used if trace quantities of sulphate and chromium are to be determined.

Polyethylene containers, in general, should be cleaned by filling with 1 mol/l nitric acid or hydrochloric acid, leaving for 1 to 2 days, followed by thorough rinsing with distilled or de-ionized water.

3.3.1.2 *For samples for determination of pesticides, herbicides and their residues* — In general, brown glass containers should be used because plastics, except polytetrafluorethylene (PTFE), may introduce interferences which can be significant if trace analyses are to be performed.

The containers should be cleaned with water and detergent, followed by thorough rinsing with distilled water, then oven dried and cooled before being rinsed with hexane or petroleum ether. Finally they should be dried with a stream of carefully purified air or nitrogen.

A continuous extraction with acetone for 12 h, followed by a hexane rinse and drying as described above, can also be used.

3.3.1.3 *For samples for microbiological analysis* — The containers shall withstand a 160°C sterilization and shall not produce or release at this temperature any chemicals which would either inhibit biological activity, induce mortality or encourage growth.

When lower sterilization temperatures are used, polycarbonate and heat resistant polypropylene containers may be used. Caps or other stoppers shall withstand the same sterilization temperatures as the containers.

Glass containers should be cleaned with water and detergent, followed by thorough rinsing with distilled water. Then they should be rinsed with nitric acid (HNO_3) followed by thorough rinsing with distilled water in order to remove heavy metals or chromate residues.

A total of 0·1 ml of a 10 percent (*m/m*) solution of sodium thiosulphate ($Na_2S_2O_3$) can be added, for every 125 ml of container capacity, before sterilization. This is to eliminate inhibition of bacteria by chlorine.

3.4 *Sample Volume* — A two-litre sample is normally sufficient for most physical and chemical analysis. However, the quantity may be varied depending upon the type of analysis, methods used etc.

3.5 *Sample Preservation* — Waste waters usually decompose rapidly at room temperature, therefore, certain tests, namely, dissolved oxygen, sulphides, residual chlorine, nitrite, *p*H, etc, should be made or fixed at site. For certain other tests, preservatives should be added immediately to individual samples of the same water or wastewater in different sampling bottles for each test. Summary of requirements for handling of samples is given in Table 1.

3.6 *Sampling Devices* — Glass or polyethylene bottles are buoyant therefore, a sufficiently heavy bracket or holder as given in Fig. 1 should be used to overcome buoyancy. The bracket should be tied with a string and lowered into canal, river or well. To collect sample from a particular depth, a sampler as given in Fig. 2 may be used. The sampler is lowered to a desired depth and its stopper is removed by means of a jerk. When the bottle is full, it cannot be stoppered and should be pulled in open condition.

3.6.1 A sampler as given in Fig. 3 should be used for sampling from 50 metres or more depth. The sampler comprises bottles open at both ends. The bottle is lowered to the desired depth in open position then closed by drop weight or messanger which slides down the supporting cord.

3.6.2 *Sub-surface sampler* — It is a device used to collect fluid samples from a bore hole at a desired depth. It is very useful in collecting water samples from geothermal boreholes and in making proper and complete geochemical study of the system underground. The design of the sampler is shown in Fig. 4. A sample vessel (*c*) is fitted at the lower end with a sample release value (*D*) and an inward flow non-return valve (*B*) at the upper end. A mild-steel shim puncture seal (*B*2) is located above, and in series with the non-return valve. A spring suspended weight fitted at its lower end with a shim seal spear, comprising the inertia mechanism (*A*), is mounted directly above the shim seal.

TABLE 1 TECHNIQUES GENERALLY SUITABLE FOR THE PRESERVATION OF SAMPLES

(*Clause* 3.5)

Sl No.	Parameters to be Studied	Type of Container	Preservation Technique	Minimum Volume, ml	Maximum Recommended Preservation Time Before Analysis	Remarks
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Acidity	P, G (B)	Refrigerate at 4°C	100	24 h	Preferably analyzed at the spot
ii)	Alkalinity	P, G (B)	Refrigerate at 4°C	100	24 h	
iii)	BOD	P, G	Cooling between 2 to 5°C and store in dark	1 000	24 h	—
iv)	Boron	P	—	200	Several months	—
v)	Carbon, organic	G (B)	Acidification to $pH<2$ wih sulphuric acid and cooling between 2 to 5°C	100	24 h	The preservation technique will depend on the method of analysis used. Test should be carried out as soon as possible. Freezing to −20°C may be used in certain cases
vi)	COD	P, G	Cooling between 2 and 5°C and store in dark Acidification to $pH<2$ Freezing to −20°C	100	As soon as possible 2 days 1 month	Acidification is particularly recommended. When the COD is due to the presence of organic materials
vii)	Carbn dioxide, total	P, G	—	100	On site	—
viii)	Chlorine dioxide	P, G	—	500	Analyse immediately	—
ix)	Chlorine, residual	P, G	—	500	Analyse immediately	Carried out on site
x)	Chlorophyll	P, G	Cooling to 4°C after filtration and freezing of residue	500	24 h 1 month	— —
xi)	Colour	P or G (Brown)	—	500	—	—
xii)	Cyanide	P, G	Add sodium hydroxide, adjust $pH>12$	500	24 h	—
xiii)	Fluoride	P	—	300	Several months if the sample is neutral	—
xiv)	Grease and oil	G, wide with calibration	Acidification to $pH<2$ extraction on site where practicable	1 000	24 h	It is recommended that, immediately after sampling, the extraction agent used in the method of analysis be added or that extraction be carried out on site
xv)	Iodide	G	Cooling to between 2 to 5°C Alkalinization to pH 8	500	24 h 1 month	Keep in dark —

(*Continued*)

TABLE 1 TECHNIQUES GENERALLY SUITABLE FOR THE PRESERVATION OF SAMPLES — *Contd*

Sl No.	Parameters to be Studied	Type of Container	Preservation Technique	Minimum Volume, ml	Maximum Recommended Preservation Time Before Aanlysis	Remarks
(1)	(2)	(3)	(4)	(5)	(6)	(7)
xvi)	Metals, dissolved	P, G	—	500	—	Separate by filtration with 0·45 µm membrane filter immediately, add reagent grade nitric acid to bring $pH<2$
xvii)	Nitrogen, ammonia	P, G	Add concentrated sulphuric acid to bring $pH<2$ and refrigerate to 2 to 5°C	500	24 h	—
xviii)	Nitrate	P, G	do	100	24 h	For certain waste-water the sample cannot be preserved and it is necessary to carry out analysis on site
xix)	Nitrite	P, G	Add mercuric chloride (40 mg/l), refrigerate to 2 to 5°C or freeze at −20°C	100	Analyse as soon as possible	—
xx)	Organic matter	P, G	Add concentrated sulphuric acid to bring the $pH<2$	500	Analyse as soon as possible	—
xxi)	Odour	G	—	500	6 h	Test shall preferably be carried out on site
xxii)	Oxygen, dissolved	P, G	—	300	Analyse as soon as possible	—
xxiii)	Ozone	—	—	1 000	On site	—
xxiv)	Pesticides, organo chloride	G	Cooling to 4°C	—	7 days	It is recommended that immediately after sampling, the extraction agent used in the method of analysis be added or that extraction be carried out on site
xxv)	Pesticide, organo-phosphorus	G	Cooling to 4°C	—	7 days	It is recommended that immediately after sampling, the extraction agent used in the method of analysis or be added or that extraction be carried out on site
xxvi)	*p*H	P, G	Transportation at a lower temperature than initial temperature	—	6 h	Analyse preferably on site
xxvii)	Phenol	G	Inhibition of biochemical oxidation by copper sulphate and acidification with phosphoric acid or alkalinization with sodium hydroxide to $pH>11$	500	24 h	The preservation technique will depend on the method of analysis to be used or type of phenol

(*Continued*)

TABLE 1 TECHNIQUES GENERALLY SUITABLE FOR THE PRESERVATION OF SAMPLES — *Contd*

Sl No.	Parameters to be Studied	Type of Container	Preservation Technique	Minimum Volume, ml	Maximum Recommended Preservation Time Before Analysis	Remarks
(1)	(2)	(3)	(4)	(5)	(6)	(7)
xxviii)	Phosphate, dissolved, inorganic	G(A)	Filtration immediately using 45 μm membrane filter and add sulphuric acid to bring *p*H <2	100	Several months	—
xxix)	Residue	P, G (B)	—	—	—	—
xxx)	Salinity	G, wax seal	Use wax seal	250	Analyse immediately	—
xxxi)	Silica	P	—	—	—	If silica is high, dilute at site with silica free water
xxxii)	Suspended and sedimentary matter	P, G	—	—	24 h	Should be carried out as soon as possible and preferably on site
xxxiii)	Sulphate	P, G	Cooling to between 2 and 5°C	—	1 week	—
xxxiv)	Sulphide	P, G	Treatment with 2 ml of 1 mol per litre of zinc acetate and alkalinization with 2 ml of 1 mol per litre sodium hydroxide	100	1 week	—
xxxv)	Sulphite	P, G	Fixing on site by addition of 1 ml of 2·5 percent (m/m) solution of EDTA per 100 ml of sample	—	1 week	—
xxxvi)	Taste	G	Refrigerate	500	—	Analyse as soon as possible
xxxvii)	Temperature	—	—	—	—	Record immediately
xxxviii)	Turbidity	P, G	Store in dark for up to 24 h	—	—	Analyse as soon as possible

Note 1 — For determinations not listed, no special requirements have been set; use glass or plastic containers, preferably refrigerate during storage and analyse as soon as possible.

Note 2 — P = plastic (polyethylene or equivalent, colourless); G = glass, G(A) or P(A) = glass, rinsed with 1 : 1 nitric acid, G(B) = glass, borosilicate, G(S) = glass; rinsed with organic solvents.

Sampler is lowered with the help of motorized wireline winch with specified speed. When it reaches the desired place/depth, it is given a jerk mechanically in a typical manner with the help of both the hands. Process is repeated five times and then sampler is pulled out. Water sample is then taken out of the sampler.

3.7 *Types of Samples*

3.7.1 *General* — Analytical data may be required to indicate the quality of water by determination of such parameters as concentrations of inorganic material, dissolved minerals or chemicals, dissolved gases, dissolved organic material, matter suspended in the water or bottom sediment at a specific time and location or over some specific time and location or over some specific time-interval.

Certain parameters, such as the concentration of dissolved gases, should be measured *in-situ*, if possible, to obtain accurate results. It is recommended that separate samples be used for chemical and biological analyses because the procedures and equipment for collection and handling are different.

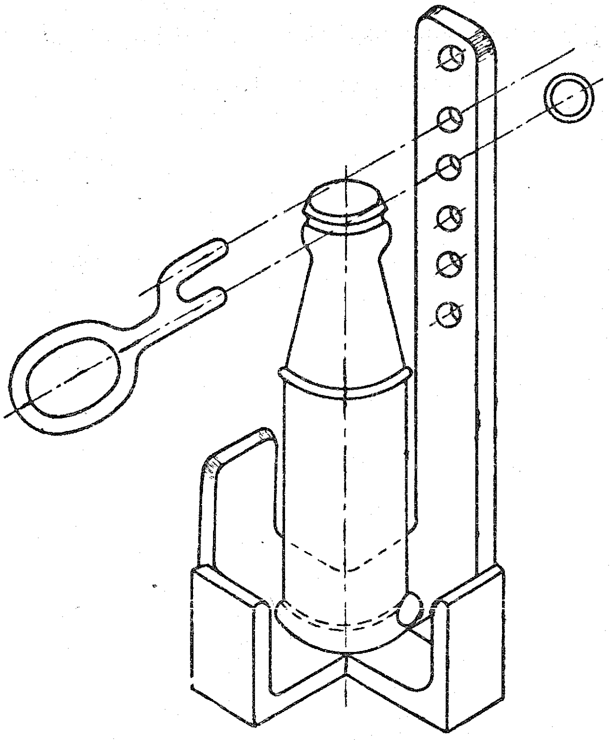


FIG. 1 SAMPLE BOTTLE HOLDER

The sampling techniques will vary according to the specific situation. The different types of sampling are described in **3.6**.

3.7.2 *Spot samples* — Spot samples are discrete samples generally collected manually, but which can be collected automatically, for waters at the surface, at specific depths and at the bottom. Each sample will normally be representative of the water quality only at the time and place taken. Automatic sampling is equivalent to a series of such samples taken on a pre-selected time or flow-interval basis.

Spot samples are useful if the flow of the water to be sampled is not uniform, if the values of the parameters of interest are not constant, and if the use of a composite sample would obscure differences between individual samples due to reaction between them.

Spot samples may also be required in investigations of the possible existence of pollution, or in surveys to indicate its extent or, in the case of automatic discrete sample collection, to determine the time of day that pollutants are present. They may also be taken prior to the establishment of a more extensive sampling programme.

The taking of spot samples may be specified for the determination of certain parameters, such as the concentration of dissolved gases, residual chlorine and soluble sulphides.

3.7.3 *Periodic samples at fixed time intervals* — These samples are taken using a timing mechanism to initiate and terminate the collection of water during a specific time-interval. A common procedure is to pump the sample during a fixed period into one or more containers, a set volume being delivered to each container.

3.7.4 *Periodic samples taken at fixed flow intervals* — These samples are utilized when variations in water quality criteria and the effluent flow rate are not inter-related. They are also categorized as flow-proportioned samples. An example would be that for each unit volume (for example, 10,000 litres) of liquid flow, a constant sample size is removed irrespective of time.

3.7.5 *Continuous samples taken at fixed flow rates (time dependent or time average)* — Samples taken by this technique contain all constituents present during a period of sampling but do not provide information about the variation of concentrations of specific parameters during the period of sampling.

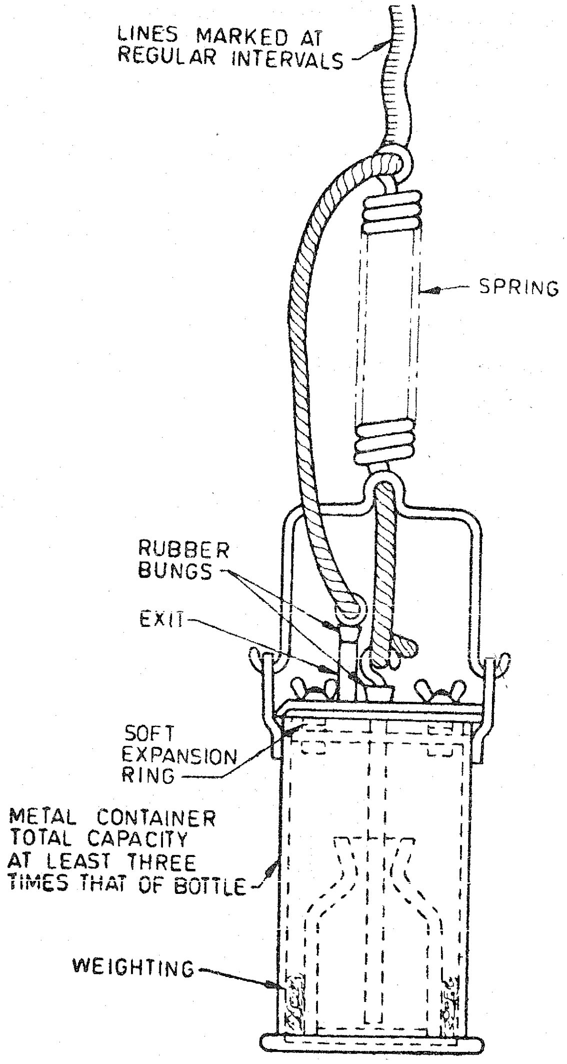

LINES MARKED AT REGULAR INTERVALS
SPRING
RUBBER BUNGS
EXIT
SOFT EXPANSION RING
METAL CONTAINER TOTAL CAPACITY AT LEAST THREE TIMES THAT OF BOTTLE
WEIGHTING

FIG. 2 IMMERSION TYPE SAMPLER USED FOR DISSOLVED GASES AND DEPTH SAMPLES

3.7.6 *Continuous samples taken at variable flow rates* (*flow dependent or proportional*)— The flow-proportional samples collected are representative of the bulk water quality. If both the flow and composition vary, flow proportional samples can reveal such variations which may not be observed by the use of spot samples. Accordingly, this is the most precise method of sampling flowing water, if both the flow rate and the concentration of pollutants of interest vary significantly.

3.7.7 *Composite samples* – Using one of the preceding techniques, samples may be obtained manually or automatically on either of two basis, that is, individual samples or composite samples, where, on either a flow, time, volume dependent or on flow basis, it is desired to mix several individual samples and reduce the cost and time for their analysis.

Composite samples provide average compositional data. Accordingly, before combining samples, it should be verified that such data is desired or that the parameter(s) of interest does not vary significantly during the sampling period.

3.8 *Transportation of Samples* – The individual wastes tend to decompose on keeping, which results in the change of composition at room temperature. The following measures should be adopted when transporting the samples from the place of sampling to the laboratory.

a) The sample should be collected in leakproof glass or plastic container;

b) Sample should be transported in an ice box keeping the temperature around 4°C;

c) Undue jerking of the samples should be avoided as this may result in coagulation of the suspended matters;

d) For bacteriological tests, samples should be handled under aspectic conditions while placing in the ice box or removing from the ice box;

e) Immediately after reaching the destination, the samples should be transferred to refrigerator;

f) A wax pencil may be used for writing details on the labels which should be protected from wetting; and

g) The sample bottles should be carefully labelled to provide the following information:

   1) Place of sampling,
   2) Time and date of sampling,
   3) Type of sampling and depth of sample,
   4) Name of the sampling staff, and
   5) Purpose of sampling.

Note — Worthy features of sampling point should also be recorded on a separate sheet and should be submitted to the laboratory along with the sample.

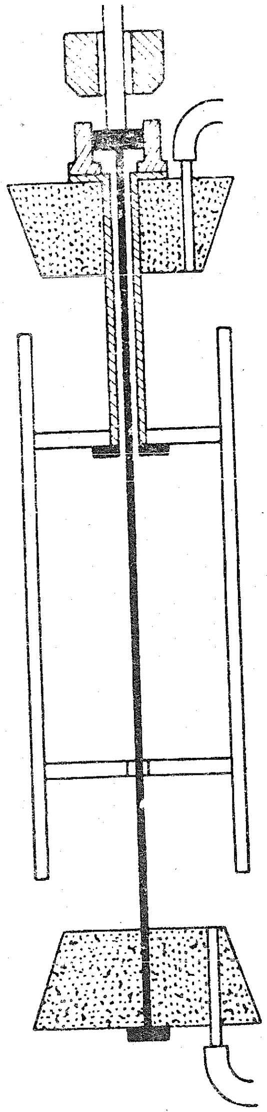


FIG. 3 KEMMERERS SAMPLER

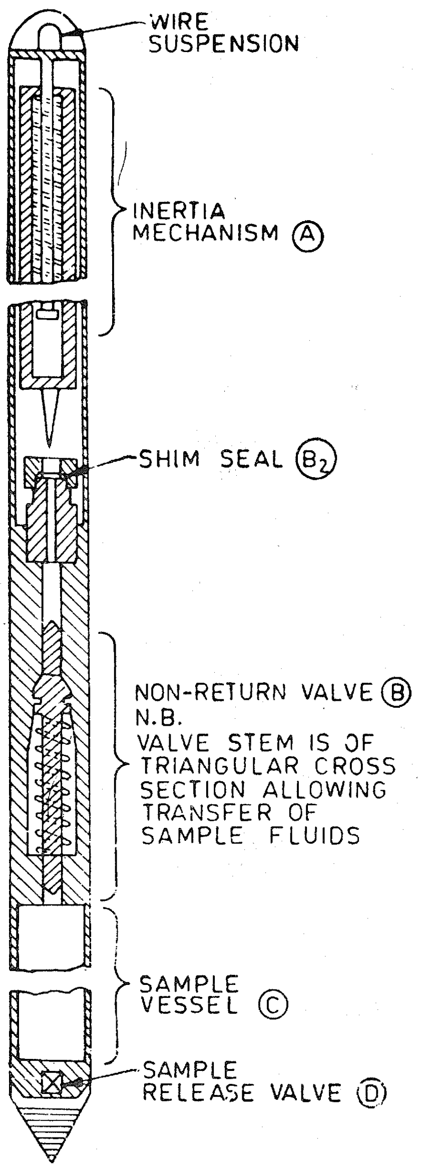
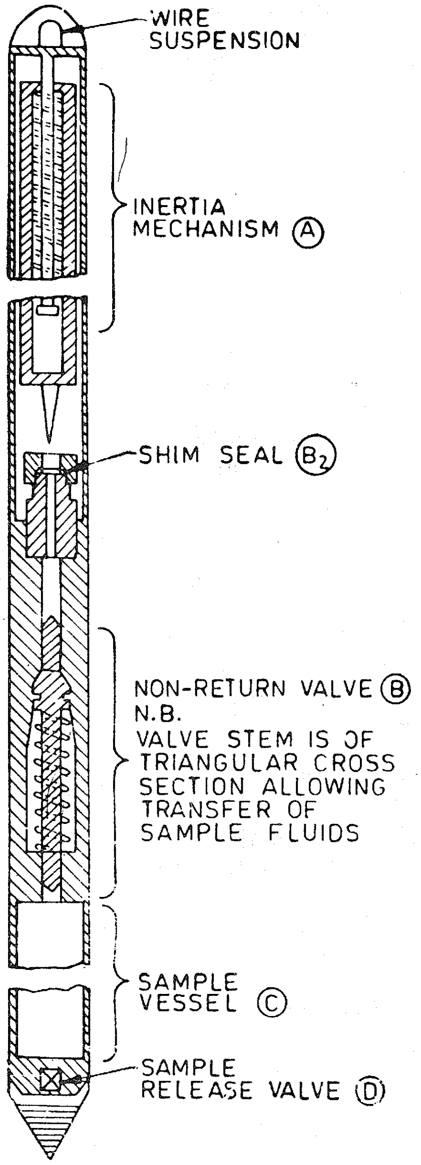


FIG. 4 KLYEN SUB-SURFACE SAMPLER

3.9 *Sampling Locations*

3.9.1 *Rivers, streams and canals* — Samples should be collected, as far as possible, from mid-stream at mid depths. Sampling too near the bank provide fictitious results. Sites should be selected preferably where marked quality changes occur and where there are important river uses such as confluences, major river discharges or abstractions. Sampling locations can be fixed by reference to significant features. In this connection use of reference maps may be helpful. The site should be reasonably accessible all the year round. Taking of samples from over the bridges is appropriate. Samples can also be taken from boats wherever feasible for rivers and lakes. Unsafe banks should be avoided. Wherever necessary, sampling should be made by a team using safety jackets. Sampling by wading, where the rivers are shallow, care being taken to collect samples upstream of the wader, who can disturb the bottom sediments.

When it is intended to monitor the effects of a discharge, both upstream and downstream sampling is necessary. Mixing of discharge with receiving water is important. A sample from 100 metres down stream of the discharge point is considered representative in case of small streams. In rivers many kilometres will be necessary. Therefore, in case of longer rivers there should be three fixed sampling locations in a cross-section (left, middle, right), the left and right one should be far enough from the bank. Sampling should extend to an appropriate distance downstream to assess effects on the river. Ideally, sample should be taken from a turbulent point. Where the flow



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